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CONTRACT NO. NAS8-33510

# FINAL REPORT OF
# INDUSTRIAL CAPABILITY TO CHEM-MILL
# ALUMINUM ALLOY 2219 IN T-37 AND T-87

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by

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## FORWORD

This document is the final report of a research study entitled "Industrial Capability to Chem-Mill Aluminum Alloy 2219 in T-37 and T-87". The work was performed under Contract No. NAS8-33510, under the technical direction of Mr. C. H. Jackson, George C. Marshall Space Flight Center.

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## 1.0 INTRODUCTION

As required by Task 1 of the referenced contract, TRITEC, Inc. established procedures and chemical baths for chem-milling aluminum alloy 2219 in T87 and T37 through a series of sample etching prior to the specimen etching called for in Task 3 through Task 5. Each specimen was numbered and its initial condition charted (Task 2) before processing. The information pertaining to the etching processes is included herein.

## 2.0 CONCLUSION

During the sample etching prior to Tasks 3 through 5, it was found that good etching results were obtained by using the "White Plastic for Porcelain Repair (toluol, xylol, and petroleum distillates)" on top of "SHIPLEY AZ-III" (Cellosolve Acetate) as resist coatings and 16° baumé ferric chloride, $FeCl_3$, as etchant. However, due to the size of the specimens (discussed in more detail in Section 4.0), the first two specimens, one each from Tasks 4 and 3, namely, specimens 37-1 and 87-1, did not give anticipated results (See Figure 4). Consequently, TRITEC, Inc. suggested to have the remaining two large size specimens, i.e., 37-2 and 87-2 reduced by 0.10m (4") to fit etching conveyor, thus allowing the specimens to be more uniformly exposed to the etchant. The results were considerably better as can be seen in Figure 4 along with the other two half size stretch formed specimens, 37-A and 37-B.

It is TRITEC's concluding opinion that chem-milling aluminum alloy in T37 and T87 can be achieved with the processes discussed in Sections 4 and 5. However, to obtain satisfactory results, it is essential that all etched portions of the specimen receive the same exposure to the etchant.

## 3.0 OBJECTIVES

As required by the referenced contract, the work to be performed by TRITEC included the following:

Task 1. Prepare chemical baths - This will be discussed in detail in Section 4.0.

Vapor Degreaser and Ultrasonic Cleaner. The solvent used, Blacotron Trichlorotrifluoroethene, is also made by Baron-Blakeslee.

Following degreasing, the aluminum was immersed for five minutes in an alkaline, satinizing pre-etch bath for removal of inorganic substances and providing a satiny finish on the material. Any presence of oil would show up as breaks in the finish at this stage. When no visible break was present, the material was then rinsed with cold water.

The pre-etch bath is a 5% caustic soda solution made by adding 0.91 Kg (32.5 ounces) of sodium hydroxide pellets into 19L (5 gallons) of water.

For removal of surface oxides, organic matters, and any small amount of oil that may remain on the material, the aluminum was further dipped in Isoprep 184 Non-Chromated Aluminum Desmutter for 3.5 minutes at room temperature, followed by copious water rinse. The desmutter, distributed by Allied-Kelite Company, was diluted to 25% by volume with water.

Although the plates were suitably clean for resist application, the deep etching (photomilling) required for this particular instance warranted the use of a conversion coating to increase resist adhesion and decrease lateral etching (undercutting). Chromate conversion coatings are recommended for aluminum. Hence, samples were dipped in a 1.5 pH solution of Iridite 14-2 for six minutes at room temperature, followed by a final cold water rinse and air drying. This completes the material preparation step.

### 4.2 Resist Coating

In anticipating the large size of specimens which could not be processed through resist coating nor Photoprinting machine, the samples were not processed through regular semi-automatic resist coating-photoprinting-developing sequence. Rather, the areas to be etched on each sample were first taped with duct tape, then manually applied the resist coating on the surrounding areas. After resist coating dried, the tapes were removed.

After a few trials with different resist coatings such as regular photo resist (Shipley AZ-III), Decon "230", and the likes, it was found that the best result was accomplished by using White Porcelain Repair on top of regular photo

resist (Shipley AZ-III). Figure 5 shows the property comparison of various resists tried.

### 4.3 Etching Process and Etchant

The TRITEC etching machine consists of a conveyor which is capable of accommodating material up to 0.56m (22") in width, an etching chamber, a double layer multiple nozzles spraying system, a 285L (75 gallon) capacity etchant reservoir, a speed control, and various indicators which monitor temperature, speed, and degrees of baumé, etc.

The etchant used is 16° baumé (diluted from 42° baumé) R.C.E. (Rapid Circuit Etch) supplied by Hunt Chemical Co. which is essentially $FeCl_3$.

#### 4.3.1 Sample Etching

Under normal etching cycle, material to be etched is put on the conveyor. The speed of the conveyor is directly coupled (synchronized) with that of spray nozzles which oscillate 60° about the fixed axis parallel to moving direction of the conveyor. The material is etched from top and bottom simultaneously while moving at constant speed inside the etching chamber. To minimize the degradation of the resist coating from etchant as well as from heat generated by the etching reaction, each small sample was run at a 13-minute cycle. The final thickness of about 0.25cm (0.1") was reached at the end of 9 cycles.

Since the spraying nozzles are located above and below conveyor, the rate of etching on each side of the material might be different due to the fact that upper ones spraying downward have higher velocity than their lower counterparts. To verify this, the first small sample piece was run with the same side facing up until the final thickness of about 0.25cm (0.1") was reached. The results not only show that the top side was upto 0.061cm (0.024")deeper than the bottom at the selected points measured, but also show up to 0.08cm (.030") variation on top side and up to 0.041cm (.017") variation on bottom side. Apparently the spray patterns were not evenly distributed.

Based on this finding, it was then decided to run the second sample at twelve 10-minute cycles (a multiple of four cycles) so that there were even cycles at each orientation 90° apart in addition to even cycles facing up and down. As a result, the maximum variation on the top and the bottom were reduced

to 0.02cm (0.08") and 0.03cm (0.012") respectively, while the maximum differential depth between top and bottom was also down from 0.061cm (0.024") to 0.028cm (0.011").

4.3.2 Specimen Etching

The etching process is discussed in the order that the specimens were prepared and etched.

4.3.2.1 Specimen 37-1

As mentioned before, the size of the specimen prohibited it from undergoing regular etching process (i.e. etching while moving). Rather it was etched under stationary conditions. As a matter of fact, the specimen had to be rested on a frame above the conveyor within the etching chamber. As a result, it was much too close to the top spray nozzles while too far away from the bottom ones. Consequently, extremely uneven etching occured. This resulted in an etched surface which was rough and sponge-looking. No meaningful data was obtained with regard to rate of etching or final thickness variation.

4.3.2.2 Specimen 87-1

Suspecting that rough finishing from specimen 37-1 might be more of a material-than-process-related problem, it was then decided to try specimen 87-1. Although sponge-looking roughness did not re-occur, the thickness variation due to stationary etching still persisted. Among the points measured, the maximum variation exceeded 4.2mm (.165"). It was concluded at that point that the only way to improve the overall result was to reduce specimens 37-2 and 87-2 from 0.61m x 0.61m (24" x 24") to 0.61m x 0.51m (24" x 20") so that they fit the conveyor.

4.3.2.3 Specimen 37-A

While the remaining two full size specimens were undergoing size reduction and pre-etch preparation, the first of the two half size specimens, 37-A, was processed through normal procedures. As expected, the result is far better than the previous two specimens in terms of thickness variation and final finishing. Figure 6 shows thickness vs. etching time for points, C, D, F, I, and J measured.

4.3.2.4 Specimen 37-2

After the size was reduced, this specimen was processed the same way as 37-A. Figure 7 shows its thickness vs. etching time. Notice that etching rate reduced to such an extent after a few cycles that a change of fresh etchant at the end of the eighth cycle was required.

4.3.2.5 Specimens 87-2 and 37-B

The last two specimens were etched concurrently to reduce overall processing time. The final results are shown in Figure 4.

4.4 Etching Results

The extremes of initial (before etching) and final (after etching) thickness variation at measured points for each specimen are tabulated in Figure 4. Notice that the first two specimens were done under stationary conditions resulting in wide final thickness variations.

## 5.0 RESIST COATING STRIPPING

The last step of chem-milling process involves removal of resist coating from the specimens and final rinsing. The process was done by brushing the metal in acetone until the surfaces were free of resist coating, then followed by a rinse of water.

## 6.0 APPENDIX

A copy of contract Attachment No.1 (Specification Sketch) is attached as Figure 11 for easy reference.

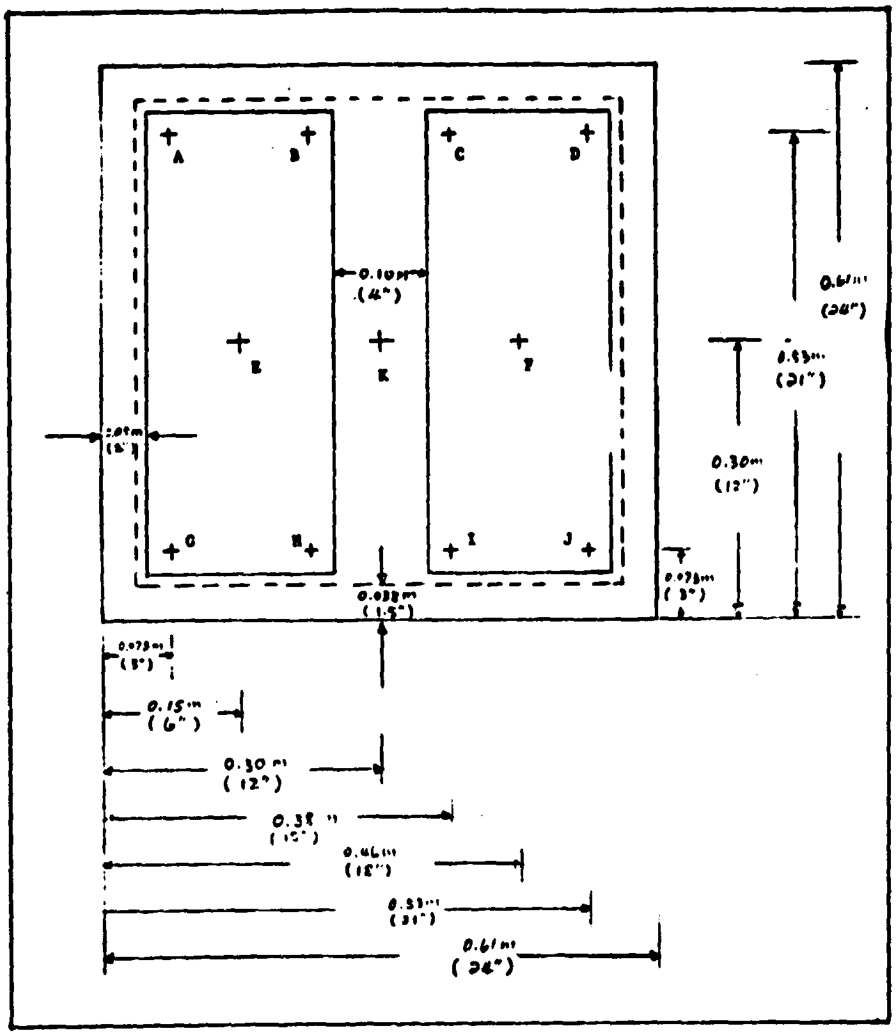


FIGURE 1: MEASUREMENT LOCATION FOR SPECIMENS 37-1 AND 87-1

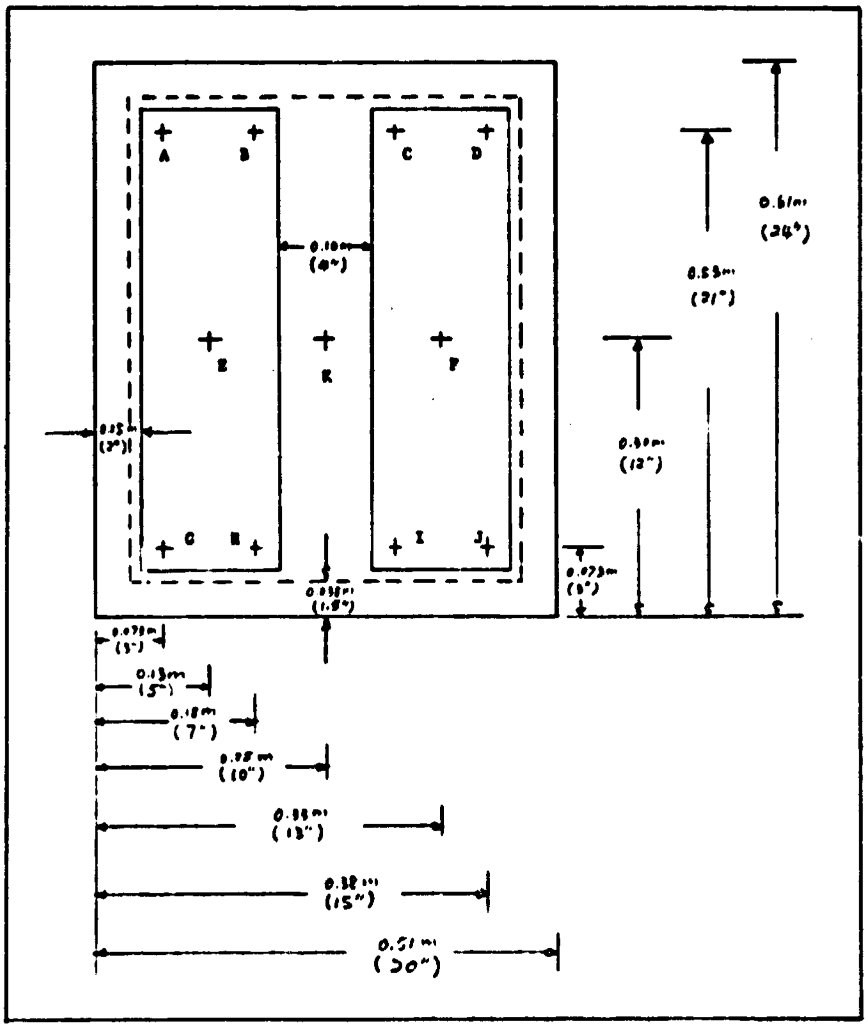


FIGURE 2: MEASUREMENT LOCATION FOR
SPECIMENS 37-2 AND 87-2

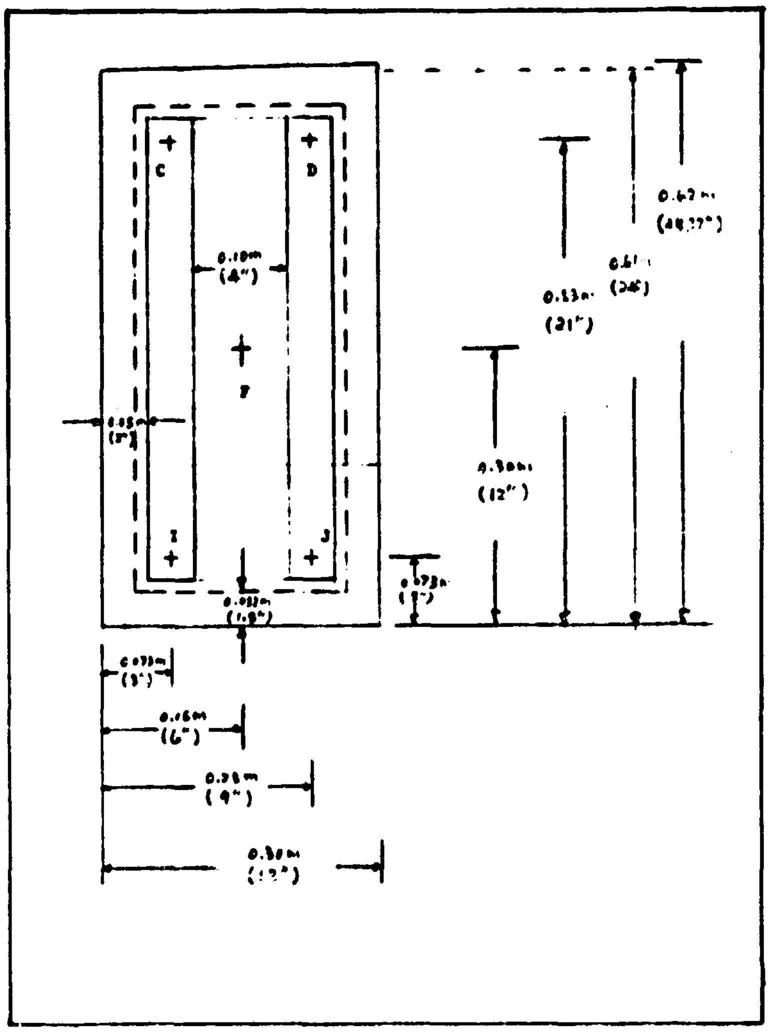


FIGURE 3: MEASUREMENT LOCATION FOR SPECIMENS 37-A AND 37-B

| ETCHING SEQUENCE | TASK NO. | SPECIMENS | INITIAL THICKNESS mm, (inch) | FINAL THICKNESS mm, (inch) | REMARKS |
|---|---|---|---|---|---|
| 1 | 4-1 | 37-1 | 12.47(0.491) + 0.05(0.002) - 0.13(0.005) | No Meaningful Measurement Can Be Obtained | Stationary Etching Due To Oversize Specimen |
| 2 | 3-1 | 87-1 | 12.47(0.491) ± 0.076(0.003) | 4.19(0.165) + 2.34(0.092) - 0.33(0.013) | Stationary Etching Due To Oversize Specimen |
| 3 | 5-1 | 37-A | 10.03(0.395) ± 0.05(0.002) | 2.82(0.111) + 0.15(0.006) - 0.25(0.010) | Specimen Size As Received |
| 4 | 4-2 | 37-2 | 12.42(0.489) + 0.03(0.001) - 0.05(0.002) | 2.44(0.096) + 0.46(0.018) - 0.28(0.011) | Specimen Size Reduced |
| 5 | 3-2 | 87-2 | 12.52(0.493) ± 0.05 0.002 0.003 | 2.84(0.112) + 0.44(0.017) - 0.58(0.023) | Specimen Size Reduced |
| 6 | 5-2 | 37-B | 9.93(0.391) + 0 - 0.025(0.001) | 2.47(0.097) + 0.22(0.009) - 0.13(0.006) | Specimen Size As Received |

FIGURE 4: TABLE OF INITIAL/FINAL THICKNESS VARIATIONS

| TYPE OF RESIST COATING TRIED | LONG DURATION ACID RESISTANCE | | | RESIST TO UNDERCUTTING | | |
|---|---|---|---|---|---|---|
| | POOR | FAIR | GOOD | POOR | FAIR | GOOD |
| Shipley AZ-111 (Cellosolve Acetate) | ★ | | | | ★ | |
| Devcon "230" (Plastic Resin and Titanium Dioxide) | ★ | | | ★ | | |
| Plastic Rubber (Toluene and Petroleum Distinuate) | | ★ | | ★ | | |
| White Plastic for Porcelain Repair (Toluol, Xylol, and Petroleum Distillates) | | ★ | | | ★ | |
| Liquid Solder (Toluene and Methylethyl Ketone) | ★ | | | | ★ | |
| White Plastic on top of Shipley AZ-111 | | | ★ | | ★ | |

FIGURE 5: PROPERTY COMPARISON OF VARIOUS RESIST COATINGS

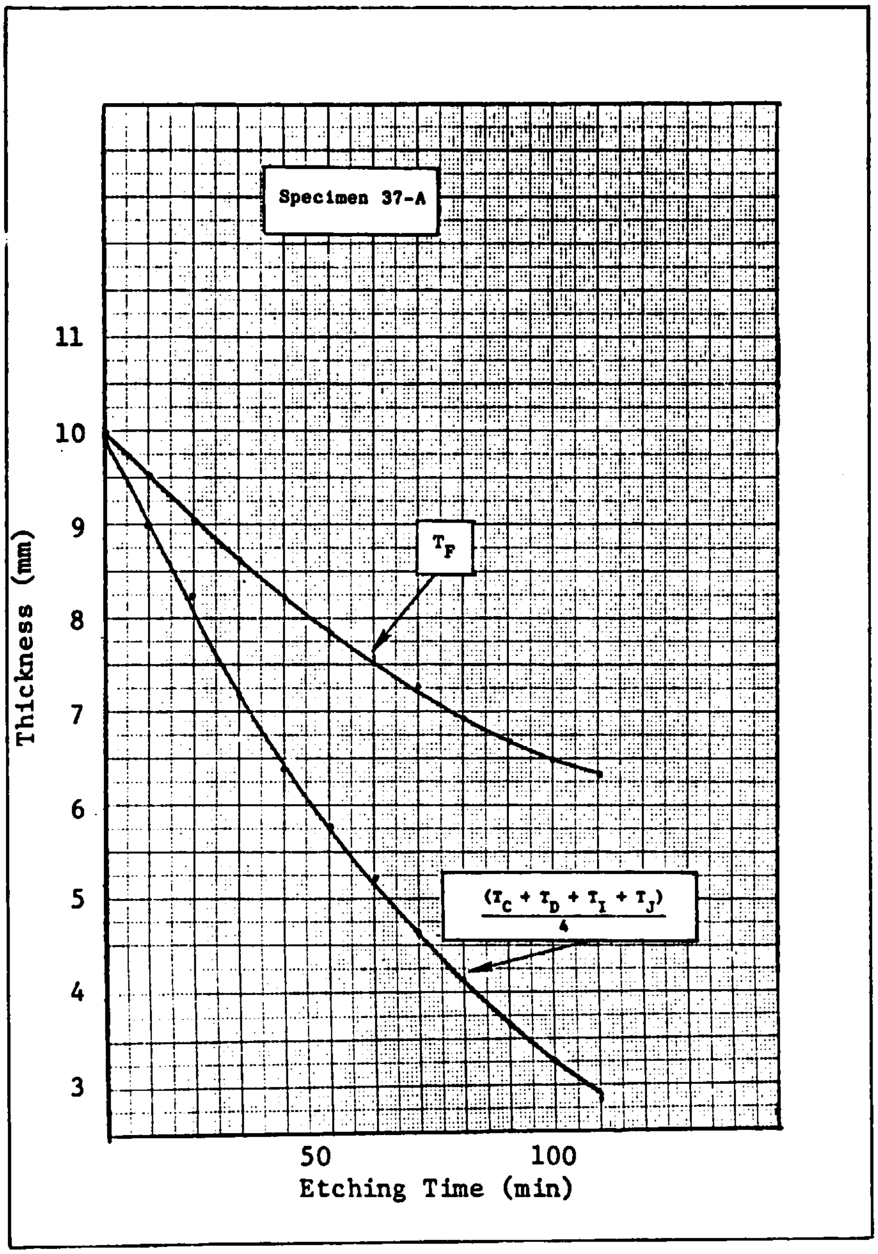


FIGURE 6. THICKNESS VS. ETCHING TIME
FOR SPECIMEN 37-A